REPORT DOCUMENTATION PAGE

Form Approved
OMB No. 0704-0188

1. REPORT DATE *(DD-MM-YYYY)*
17 March 2003

2. REPORT TYPE
Technical Paper

3. DATES COVERED *(From - To)*

4. TITLE AND SUBTITLE

Assessment of Multiple Scattering Errors of Laser Diffraction Insturments

5a. CONTRACT NUMBER

5b. GRANT NUMBER

5c. PROGRAM ELEMENT NUMBER

6. AUTHOR(S)

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5d. PROJECT NUMBER
3058

5e. TASK NUMBER
RF9A

5f. WORK UNIT NUMBER

7. PERFORMING ORGANIZATION NAME(S) AND ADDRESS(ES)

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8. PERFORMING ORGANIZATION REPORT NUMBER

AFRL-PR-ED-TP-2003-067

9. SPONSORING / MONITORING AGENCY NAME(S) AND ADDRESS(ES)

Air Force Research Laboratory (AFMC)
AFRL/PRS
5 Pollux Drive
Edwards AFB CA 93524-7048

10. SPONSOR/MONITOR'S ACRONYM(S)

11. SPONSOR/MONITOR'S NUMBER(S)
AFRL-PR-ED-TP-2003-067

12. DISTRIBUTION / AVAILABILITY STATEMENT

Approved for public release; distribution unlimited.

13. SUPPLEMENTARY NOTES

14. ABSTRACT

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20031003 096

15. SUBJECT TERMS

16. SECURITY CLASSIFICATION OF:			17. LIMITATION OF ABSTRACT	18. NUMBER OF PAGES	19a. NAME OF RESPONSIBLE PERSON
					Sheila Benner
a. REPORT	**b. ABSTRACT**	**c. THIS PAGE**	A		**19b. TELEPHONE NUMBER** *(include area code)*
Unclassified	Unclassified	Unclassified			(661) 275-5693

FILE

MEMORANDUM FOR PRS (In-House Publication)

FROM: PROI (STINFO) 17 Mar 2003

Strakey
55673

SUBJECT: Authorization for Release of Technical Information, Control Number: **AFRL-PR-ED-TP-2003-067**
Peter A. Strakey, "Assessment of Multiple Scattering Errors of Laser Diffraction Instruments"

Talley

International Conference on Liquid Atomization and Spray Systems **(Statement A)**
(Sorento, Italy, 13-18 Jul 2003) (<u>Deadline: 15 Apr 2003</u>)

Assessment of Multiple Scattering Errors of Laser Diffraction Instruments

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Abstract

The accuracy of two commercial laser diffraction instruments was compared under conditions of multiple scattering designed to simulate the high droplet number densities encountered in liquid propellant rocket combustors. Both instruments employ correction factors to account for multiple scattering at transmission levels down to about 2%. The instrument accuracy was compared in terms of several mean moment diameters as well as the standard deviation of the measured distributions. Results show that the newer instrument with a more recently developed statistical approach to correcting for multiple scattering errors produced significantly greater accuracy than the older instrument that employs a more limited type of analytical correction scheme. The statistical correction scheme resulted in an accuracy of about +/- 10% for the volume-weighted volume mean diameter, D_{43} down to a transmission of about 2%, while the analytical correction approach resulted in an under-estimation of D_{43} by as much as 45% at a transmission of 2%. With the statistical correction, reasonable accuracy was obtained at a transmission as low as 1% and was limited by the signal-to-noise ratio of the detector.

Introduction

Characterization of high-pressure rocket injector sprays often involves using commercial laser-based diagnostic instruments to measure droplet size in an environment well beyond the intended range of the instruments. Even in cold-flow measurements, this environment often involves very large number densities of droplets ($N > 10^4$ cc^{-1}) as well as refractive index gradients in the surrounding gas. As a result, optical attenuation and multiple scattering of the probe beam(s) and scattering signal is often the limiting factor in the ability to extract useable data.

Two of the most widely used diagnostic techniques to measure droplet size in a spray are phase Doppler interferometry (PDI) and laser diffraction. While PDI is often the instrument of choice, it is limited to the measurement of spherical droplets. Liquid bi-propellant rocket injectors often generate large, non-spherical droplets as a result of the relatively low injection velocities and high chamber back-pressures which create high deformation stresses on the droplet.

Laser diffraction instruments are capable of characterizing large non-spherical droplets and typically report a droplet diameter that is the spherical equivalent to the cross-sectional area of the non-spherical droplet.[1] While this is not necessarily ideal, it does allow for characterization of the spray. Since the laser diffraction technique is a line-of-sight ensemble scattering technique, it is prone to errors associated with multiple scattering.[2-7]

The goal of this study is to assess the capability and limitations of the laser diffraction technique in dense sprays. In this study, two commercial laser-diffraction instruments were tested. The first was a Malvern 2600c Master Sizer system and the second was a Malvern SprayTech instrument. Both instruments are similar in that they yield a measure of the droplet size distribution by inverting the light scattering information collected with a Fourier transform lens and a multi-ring photodetector. The 2600c averages the scattered light for a pre-set period of time, after which the distribution is calculated from Fraunhofer diffraction theory using a least-squares fitting routine. Previous studies with similar systems have shown that multiple scattering effects become important when the transmission of the laser beam is less than about 50%. The 2600c incorporates an analytical correction factor developed by Felton *et al.* to correct the size distribution using a two-parameter size distribution function.[3] This function is user selectable as either a

Rosin-Rammler or log-normal size distribution. With the multiple scattering correction, the 2600c instrument has been reported to be accurate at transmissions as low as 2.5%.

The SprayTech instrument is different in several ways. First, it collects a temporally resolved measurement of the light scattering distribution, then calculates the droplet size distribution using a pre-generated look-up table based on Lorenz-Mie scattering theory. A large number of measurements can be made over a period of time with the resulting droplet size distributions of each individual measurement being averaged to form a time-averaged distribution. The second notable difference is that the multiple-scattering correction scheme does not require that the distribution be fit to a mathematically described distribution; the shape of the distribution can be completely arbitrary. The SprayTech instrument is reported to be accurate at transmissions as low as 2%.

Experimental Setup

In order to assess the accuracy and limitations of the instruments described above, it was necessary to simulate the dense spray environment with a two-phase medium of known particle number density and distribution. This was accomplished using a dispersion of solid, spherical polystyrene microspheres (Duke Scientific 4000 Series Polymer Microspheres) and distilled water in a magnetically stirred glass test cell. The microspheres are transparent in the visible spectrum and have an index of refraction of 1.55 in air, resulting in an index of refraction of 1.16 in water. The square test cell had a path length of 37 mm and was placed in the beam path with the opposing windows canted about 2° from normal with respect to the laser beam propagation direction. This ensured that reflections from the windows were not incident on the detector. A schematic of the experimental setup is provided in Figure 1.

Separate experiments were conducted with each instrument using both monodispersed microspheres at concentrations ranging from 90% to 1% transmission and sizes ranging from 30 μm to 650 μm. Table 1 contains the specified microsphere diameter and standard deviation of the size distribution for each sample. This data is provided by the manufacturer and is NIST traceable. Assessment of the accuracy of the instruments under conditions of multiple scattering was carried out by first making a background measurement with the glass test cell filled with distilled water. The microspheres were then added and continuously stirred until a transmission of about 90% was obtained. Data was then collected for five seconds while the test cell was being continuously stirred. The averaged data was then processed to yield an average particle size distribution. Additional microspheres were then added and the process repeated until a transmission of about 1% was obtained.

Experiments were also conducted with polydispersed mixtures of beads over the same range of concentrations and sizes. The polydispersed mixtures consisted of six different bead sizes in relative concentrations that approximated a lognormal distribution. The polydispersed mixtures were formed by starting with pure water and then subsequently adding beads of the smallest size class (30 μm) until a pre-determined transmission was obtained. The transmission was determined by assuming that the relationship between transmission and number density could be described by Beer's law: $I/I_o = exp(-kNL)$, where the extinction cross section, k, was taken to be twice the cross sectional area of the sphere $(k = 2\pi D^2/4)$. N is the particle number density and L is the path length through the cell. This process was repeated with each size class using the relationship between total transmission and number density of each size class: $I/I_o=exp(-L\Sigma k_iN_i)$ until the final concentration was obtained with a resulting transmission of about 1%. The mixtures were continuously stirred throughout the preparation and measurement process. Data was collected with each instrument for a period of five seconds after which the data was processed and averaged over the collection time. The mixtures were then diluted by removing one-third of the mixture with a syringe and replacing the removed fraction with distilled water. In this fashion the overall concentration could be reduced without affecting the size distribution.

The repeatability of this procedure was tested by performing the process several times and measuring the standard deviation in the mean particle size measured by the 2600c instrument. The standard deviation in D_{43} for all of the runs was found to be less than 6% of the mean. Also, measurements were taken at several locations within the test cell to determine if the stirring bar was creating a striation in the microsphere mixture within the cell. The variation in mean size across the cell was found to be about the same as the

variation in several sequential measurements taken at one location in the cell, which was 2 % of the mean. In an effort to check for possible systematic errors introduced by the dilution procedure, a separate set of experiments was conducted in which the concentrated mixtures were circulated through the test cell using a small centrifugal pump. The bead mixture was continuously pumped out of the test cell, then through a separate reservoir into which distilled water could be added to dilute the mixture before being pumped back into the test cell. In this fashion, the number density could be reduced through dilution without the actual removal of any of the microspheres. The maximum variation in D_{43} between this test procedure and the dilution procedure described earlier was found to be 8%. Thus, the maximum error in D_{43} associated with the experimental technique itself is believed to be +/- 8%.

Both instruments were optically configured to cover a similar range of particle size measurement. The 2600c was outfitted with either a 600 mm lens that provided a measurable size range of 11.6 to 1128 µm or a 300 mm lens yielding a size range of 5.8 to 564 µm. The Spraytech was configured with either a 450 mm lens covering the size range of 8.6 to 1041 µm or a 200 mm lens covering the range of 3.8 to 463 µm.

Results and Discussion

Monodispersed Microspheres

Results from the monodispersed experiments are presented as a percent error in the volume-weighted volume mean diameter, D_{43} from the actual size. D_{43} was chosen as a representative indicator of accuracy because both instruments use a process of inverting the light scattering data to obtain a particle volume distribution and are thus geared toward providing maximum accuracy in a volume mean diameter. Figures 2 and 3 are plots of percent error in measured D_{43} as a function of light transmission through the test cell for the 2600c and Spraytech instruments respectively.

The 2600c (Figure 2) provided reasonable accuracy at transmissions down to 2% with a general trend of under-measuring the particle size (negative error) as the particle number density was increased. This is consistent with the fundamental problem of multiple scattering in which the overall angle of light scattering increases with each scattering event. Since scattering angle is inversely proportional to particle size, the increase in overall scattering angle results in a smaller measured size. The multiple scattering correction algorithm used by this instrument did not fully compensate for this effect; however, it did greatly improve the accuracy over using no correction at all as will be discussed later.

The Spraytech instrument (Figure 3) produced an error that was generally less than the 2600c instrument. Also, the correction scheme used by this instrument appears to be less sensitive to the particle number density as evidenced by the relatively flat curves in Fig. 3. This can be explained by the differences in the multiple scattering correction schemes used by the two instruments. The 2600c uses an analytical correction model developed by Felton *et al.* that divides the light path into series of slices of equal attenuation.[3] Their assumptions included: each slice scatters 10% of the incident light; only half of the scattered light is forward scattered and there is no multiple scattering within the slice. These assumptions are not necessarily true in many cases and limit the applicability of this approach. This approach does however allow the correction to be applied at very little computational expense. The multiple scattering correction scheme used in the Spraytech instrument is based on a statistical approach proposed by Hirleman.[8] In this approach a scattering redistribution function is calculated based on the probability of each scattering event (*ie.* single scatter, double scatter, etc.). The probability of each event is calculated from Poisson statistics and the light reaching the detector is assumed to be the summation of all of the significant scattering events. This approach requires fewer assumptions at the expense of increased computational complexity. This approach has been made possible by the tremendous advancements in low-cost computational power in the past twenty years.[9,10]

To show the importance of correcting for multiple scattering, the data for several of the tests conducted with the Spraytech instrument was processed without the multiple scattering correction. This data is shown in Figure 4 along with the same data processed with the correction. Without the multiple scattering correction a significant decrease in measured size is found with increasing number density (decreasing

transmission). This is due to the increase in scattering angle with each multiple scattering event as discussed earlier.

Polydispersed Results

The polydispersed mixtures, as described earlier, were formulated to follow a lognormal distribution. This was done for two reasons. First, the physics of atomization typically results in a spray that follows some sort of mathematically described distribution. Second, the multiple scattering correction algorithm used by the 2600c instrument requires that the distribution be fit to a two-parameter mathematically described distribution. The range of volume-weighted volume mean diameters studied here is characteristic of the sizes produced by like-on-like bi-propellant liquid rocket injectors. The actual mean diameters of each for the twelve mixtures was determined by measuring the transmission during the preparation of the mixtures and are given in Table 2.

An example of a typical distribution (*Run 5*) along with the lognormal "target" volume distribution is shown in Figure 5. This particular distribution can be represented by the geometric mean diameter, D_g, and the width of the distribution, σ_g.

$$n(D) = \frac{N}{(2\pi)^{1/2} D \ln \sigma_g} \exp\left[-\frac{(\ln D - \ln D_g)^2}{2\ln^2 \sigma_g}\right] \qquad \text{eqn. (1)}$$

Where $n(D)$ is the number density of size D, and N is the total number density of particles. The moment mean diameters of the distribution can be described with the general equation;

$$D_{pq} = \frac{\int_0^\infty n(D) D^p dD}{\int_0^\infty n(D) D^q dD} \qquad \text{eqn. (2)}$$

Three diameters were chosen here to represent the measured averages of the distributions. They are the volume-weighted volume mean diameter, D_{43}, the Sauter mean diameter, D_{32}, and the arithmetic mean diameter, D_{10}. These averages represent a broad range of mean diameters across both the volume and number distributions and should be sufficient to characterize the instrument accuracy. Along with the mean diameter in Table 2 is the standard deviation of the volume distribution normalized by the mean, σ/D_{43} and also the total particle number density, N, for the case of minimum transmittance. The standard deviation for the mixtures as well as that reported for the measured data is defined for the volume distribution as;

$$\sigma = \left[\frac{\sum n(D) D^3 (D - D_{43})^2}{\sum n(D) D^3}\right]^{1/2} \qquad \text{eqn. (3)}$$

Figure 6 contains plots of measurement error, expressed as a percentage of D_{43} as a function of transmission for each instrument. In both cases, the multiple scattering correction option has been used. The Spraytech instrument (Fig. 6(a)) showed much better accuracy over the range of transmissions studied here. The instrument was accurate to within +/- 10% in the transmission range of 90% to 2%, which is the stated lower range of the instrument. The instrument produced reasonably good results even at a transmission of 1%. The minimum error occurred at about 60% transmission for all the distributions measured. The reason for this is that maximum signal to noise occurs at around 50% transmission, where multiple scattering effects are negligible and the signal strength reaching the detector is a maximum.[10] At transmissions above 50% the scattered light level begins to drop rapidly and some error is introduced into the measurement by the presence of background noise. At transmissions below 50%, the scattered light

signal also begins to drop off, introducing noise error as well as that caused by multiple scattering. The multiple scattering algorithm does seem to do an excellent job of mitigating multiple scattering errors, and the minimum useable transmission is really determined by the minimum useable signal reaching the detector (around 1%). The results from the 2600c instrument were not as encouraging. Even with the multiple scattering correction, the measured D_{43} began to drop significantly below a transmission of about 10%. At a transmission of 2% the measured D_{43} was found to be less than the actual D_{43} by as much as 45%. The general trend of measured average diameter decreasing with decreasing transmission (increasing multiple scattering probability) is consistent with the mechanism of increasing scattering angle with each multiple scattering event as discussed earlier. This result is qualitatively consistent with the findings of Dodge who found that even with the correction, the instrument underestimated mean size by as much as 20% at a transmission of about 5%.[11]

Figure 7 shows the error in D_{32} as a function of transmission. Both the trends and magnitude of error are consistent with those found for D_{43} (Fig. 6). Figure 8 shows the error in D_{10} as a function of transmission. Notice that the measured error for the Spraytech instrument increased dramatically for this smaller moment mean diameter. The reason for this lies in the inversion algorithm used by the SprayTech instrument. The measured light scattering distribution across the 31 rings is fit to a volume distribution using a non-linear inversion technique. With this approach the error will generally be minimized where the volume distribution and light scattering intensity is at a maximum. The largest errors will occur in the "wings" of the volume distribution where volume is at a minimum. The arithmetic mean diameter, D_{10}, is representative of the number distribution, which lies in the lower wing of the volume distribution where there is a significant amount of error. In transformation to the number distribution the error is raised to the third power and hence the error in measured D_{10} is quite large.

The 2600c instrument (Fig. 8b) did not show as large an increase in error from the larger moment diameters. This is because the inversion from light scattering distribution to particle size distribution was done assuming a log-normal distribution for the particle size distribution. This effectively forces the wings of the volume distribution to zero and minimizes fitting errors in this part of the distribution. Several of the data points collected with the 2600c instruments were analyzed with the model-independent inversion option which makes no assumptions on the shape of the particle size distribution (similar to the SprayTech). In this case large negative errors were observed as was the case with the SprayTech instrument. The 2600c does not, however, allow for a multiple scattering correction with the model-independent analysis, and large errors were found for all of the moment diameters at low transmission levels. It was thus decided to use the log-normal model in all of the 2600c data reduction. In general it is not recommended that either of these instruments be used for reporting the smaller moment diameters, such as D_{10} due to the large uncertainty introduced during the analysis.

Figure 9 presents a summary plot of percent error for each of the three moment diameters analyzed here. In each plot a third order polynomial curve fit of average error for all of the data is plotted along with the 95% prediction interval for each curve. The prediction interval describes the range of predicted error to within a 95% degree of confidence.

Aside from the mean diameters, another parameter of interest with respect to sprays is the width of the distribution. A common parameter characterizing the width is the standard deviation, σ. The standard deviation of the volume distribution is defined as Equation 3. A plot of percent error of the measured σ for all of the data is presented in Figure 10. The Spraytech instrument, which makes no assumptions about the shape of the distribution clearly provided much better accuracy (+/- 30%) with respect to σ. For the 2600c instrument the error in σ was significantly larger. Although the prepared distributions were lognormal in nature and the data analysis with the 2600c instrument was conducted with the lognormal model option, the discrete nature of the polydispersed bead mixtures could explain some of the relatively large errors measured with this instrument. As seen in Fig. 5, the wings of the lognormal distribution extend well past the actual bounds of the discrete distribution.

A comparison in the ability of the instruments to capture the actual distribution at high and low transmission is presented in Figure 11. Note that the absolute magnitude between the actual and measured distributions is somewhat different. The abscissa of the plots is expressed as the fraction of volume in each

bin and the actual distribution consists of six discrete size classes, or "bins", while the measured distributions are sorted into about 30 bins. For the 2600c instrument, which is using a lognormal fit to the distribution, the shift towards smaller sizes is clearly evident in the figure. The SprayTech instrument does show some smearing of the peaks at the lower transmission, but accuracy is still preserved in the mean moment diameter, D_{43}.

Summary and Conclusions

For the monodispersed cases, the accuracy in D_{43} for the SprayTech instrument was +/- 6% at transmissions down to about 1%. The 2600c instrument was somewhat less accurate at +/- 10% at transmissions down to 2%. Most of the difference in accuracy is believed to be due to the more robust multiple scattering correction algorithm employed by the SprayTech instrument.

For the polydispersed cases, the accuracy in D_{43} for the SprayTech instrument was about +/- 10% at transmissions down to 2% with slightly larger (negative) errors down to 1% transmission. The 2600c instrument showed larger errors with a significant under-estimation of D_{43} at transmissions below 10%. Errors as large as 45% were observed at a transmission of 2%. Similar results were found for D_{32}.

The error in measured D_{10} for both instruments was much larger than for D_{43} and is believed to be due to error in the inversion algorithms which fit the measured light scattering distribution to a volume weighted particle distribution. It is not recommended that either instrument be used to report mean diameters characteristic of the diameter weighted distribution.

In terms of dense spray measurements, the limiting factor for the 2600c instrument appears to be the multiple scattering correction which is reliable only to about 10% transmission. For the SprayTech instrument, the accuracy is limited by signal-to-noise errors that become significant below about 2% transmission. The instrument is still useable however down to a transmission of about 1%.

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Table 1 : Polystyrene sphere sizes.

Nominal Diameter (μm)	Actual Diameter (μm)	Standard Deviation (μm)
30	30.1 +/- 0.22	0.45
60	59.8 +/- 1.0	2.0
100	100 +/- 1.8	1.6
200	200 +/- 4.0	5.2
400	400 +/- 8.0	15.2
650	646 +/- 13	24.8

Table 2: Prepared polydisperse distributions.

Run	Instrument	Lens (mm)	D_{43}(μm)	D_{32}(μm)	D_{10}(μm)	σ/D_{43}	N (cc^{-1})
1	2600c	600	205	143	73	.649	$9.3e^3$
2	2600c	600	237	164	84	.641	$6.9e^3$
3	2600c	600	208	154	86	.632	$7.1e^3$
4	2600c	300	76	70	49	.286	$2.1e^4$
5	2600c	300	189	158	112	.349	$4.6e^3$
6	2600c	300	315	275	192	.322	$1.5e^3$
7	SprayTech	200	76	70	49	.286	$2.1e^4$
8	SprayTech	200	189	158	112	.349	$4.6e^3$
9	SprayTech	450	315	275	191	.322	$1.5e^3$
10	SprayTech	450	462	388	142	.386	$2.2e^3$
11	SprayTech	450	115	94	69	.463	$1.5e^4$
12	SprayTech	450	338	185	42	.571	$2.9e^4$

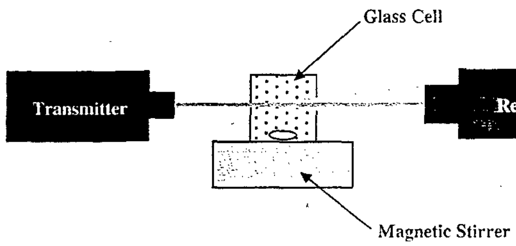

Figure 1 : Experimental setup.

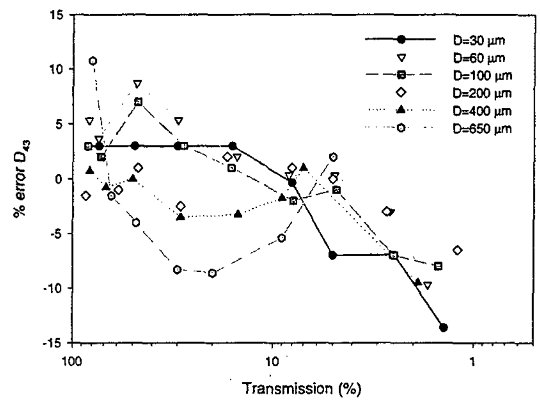

Figure 2: Percent error in measured D_{43} versus transmission for the 2600c instrument, monodispersed results.

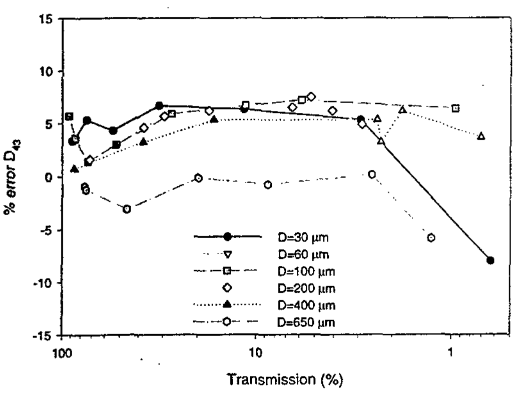

Figure 3: Percent error in measured D_{43} versus transmission for the Spraytech instrument, monodispersed results.

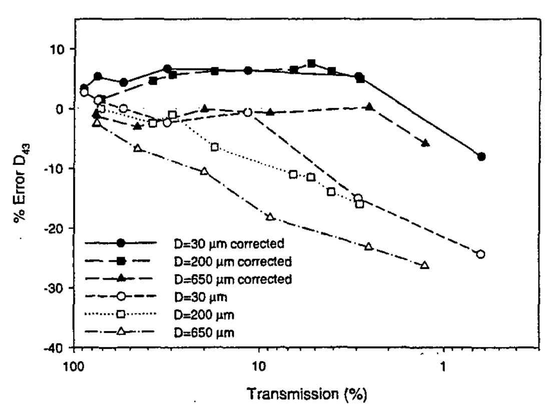

Figure 4: Percent error in measured D_{43} versus transmission for the Spraytech instrument, monodispersed results, with and without multiple scattering correction.

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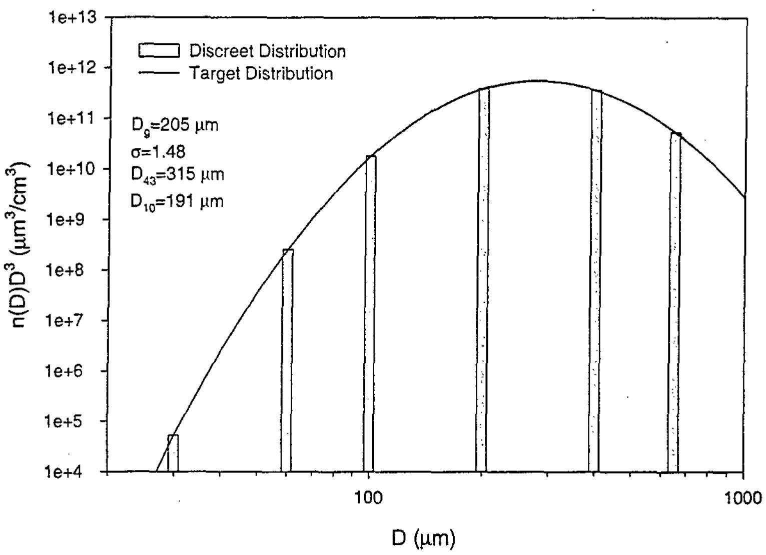

Figure 5: Typical volume distribution for the polydispersed mixtures showing both the "target" lognormal distribution and discrete distribution of polystyrene beams.

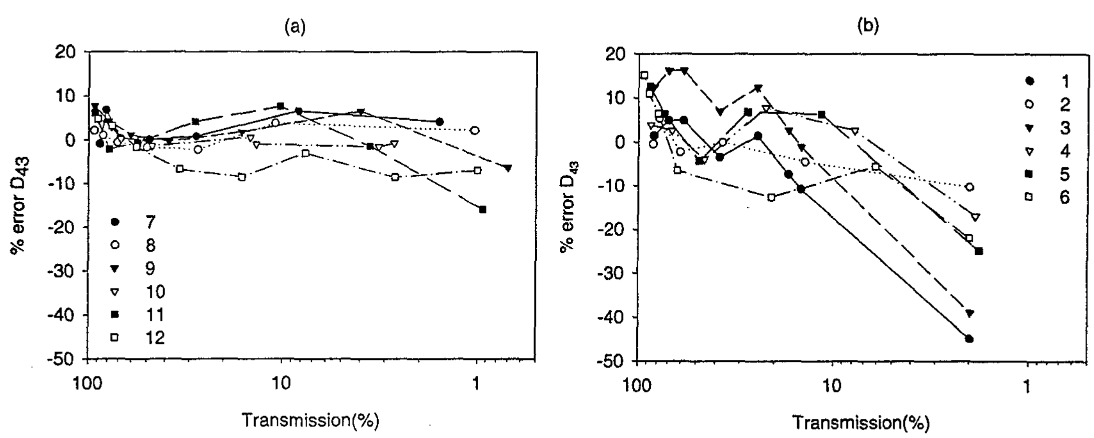

Figure 6: Percent error in measured D_{43} versus transmission for (a) SprayTech and (b) 2600c instruments for conditions listed in Table 2.

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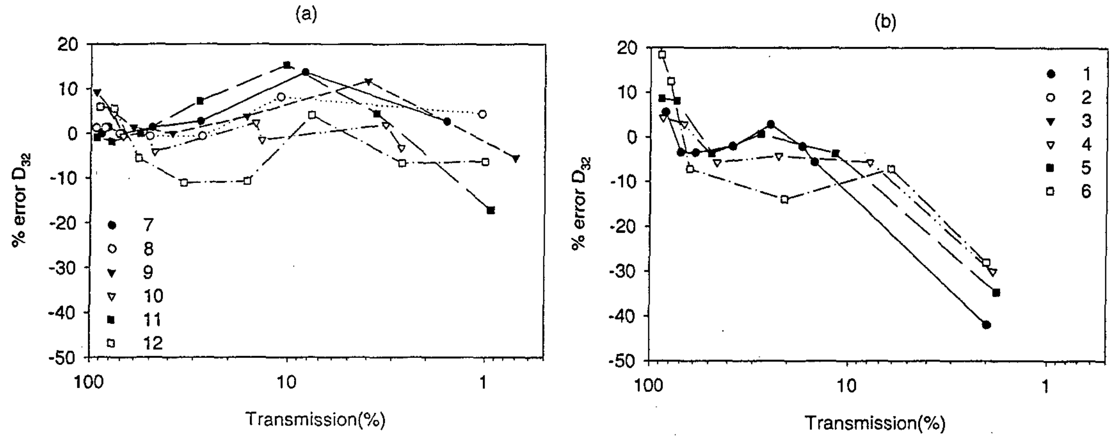

Figure 7: Percent error in measured D_{32} versus transmission for (a) SprayTech and (b) 2600c instruments for conditions listed in Table 2.

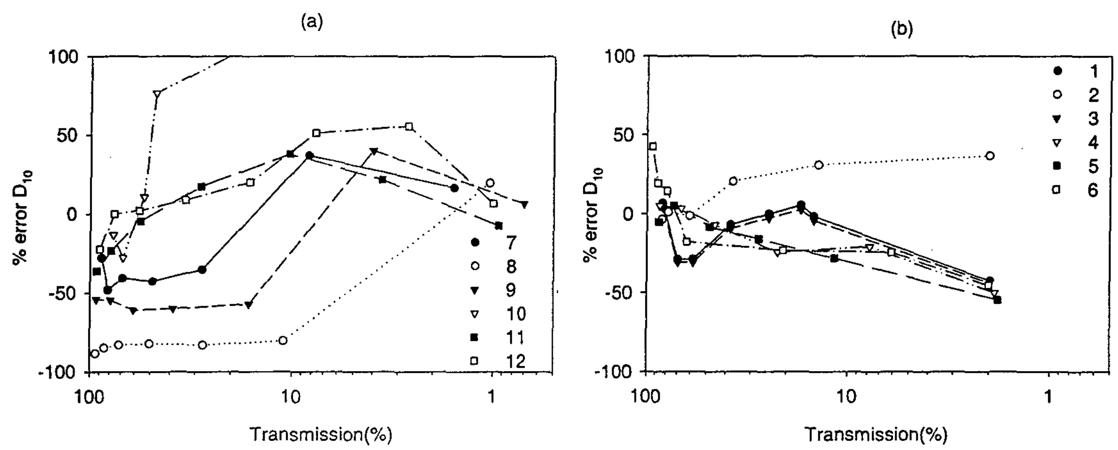

Figure 8: Percent error in measured D_{10} versus transmission for (a) SprayTech and (b) 2600c instruments for conditions listed in Table 2.

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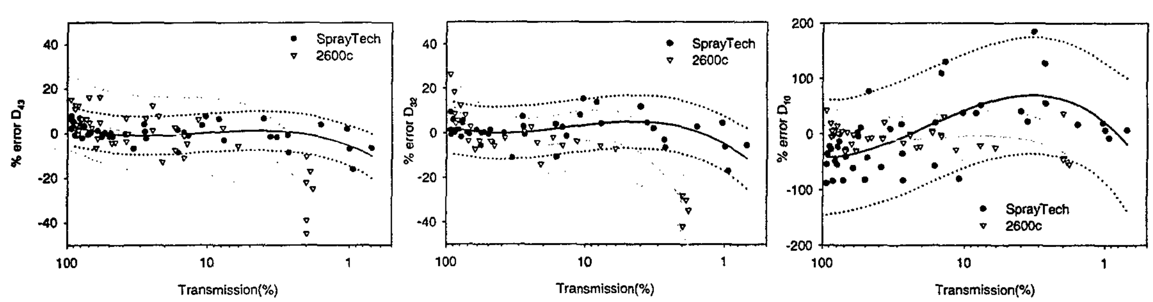

Figure 9: Summary plots of percent error versus transmission with polynomial curve fits to average error and 95% prediction intervals for both instruments for all data in Table 2.

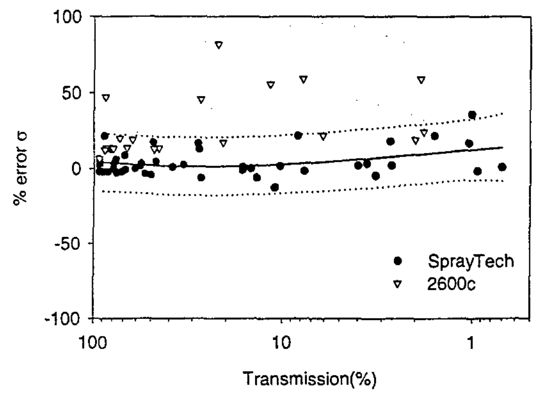

Figure 10: Percent error in standard deviation of volume distribution versus transmission for both instruments for all data in Table 2.

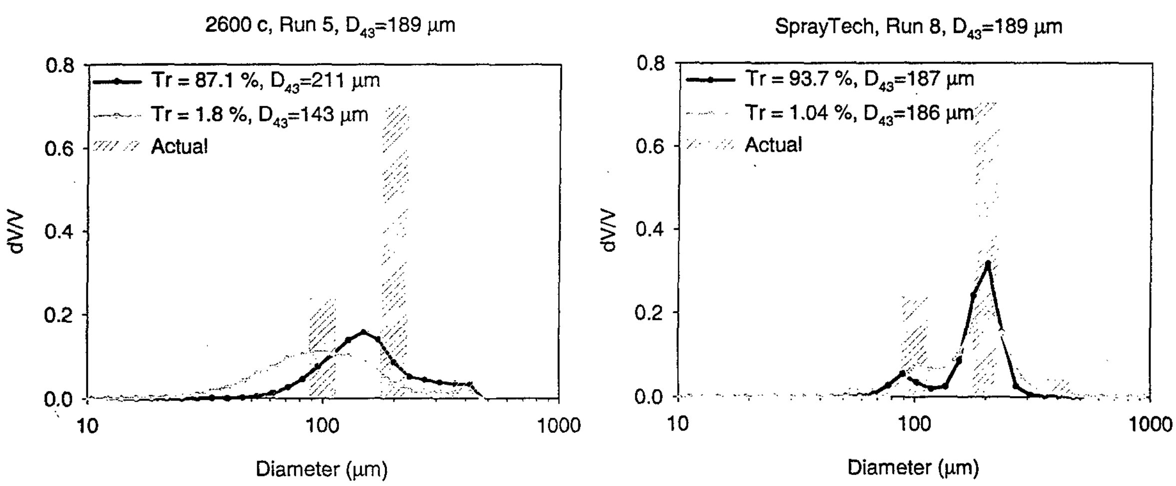

Figure 11: Measured volume distributions (percent in bin) at high and low transmission and actual distribution for both 2600c and SprayTech instruments.

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